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IS 4027-8 (1991): Methods of chemical analysis of bronzes, Part 8: Determination of iron [MTD 8: Copper and Copper Alloys]



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भारतीय मानक

कांसे के रासायनिक विश्लेषण की पद्धतियाँ

भाग 8 लोहा ज्ञात करना

(पहला पुनरीक्षण)

Indian Standard

**METHODS OF CHEMICAL ANALYSIS
OF BRONZES**

PART 8 DETERMINATION OF IRON

(First Revision)

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BUREAU OF INDIAN STANDARDS
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FOREWORD

This Indian Standard (Part 8) (First Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Methods of Chemical Analysis of Non-ferrous Metals Sectional Committee had been approved by the Metallurgical Engineering Division Council.

IS 4027 first published in 1967, covered determination of copper, lead, tin, manganese, phosphorus, nickel, iron, silicon, aluminium, zinc and antimony in bronzes. While reviewing this standard, the Sectional Committee decided that it is convenient to revise this standard in series of parts, which on publication will supersede the relevant method for determination given in IS 4027 : 1967 'Methods of chemical analysis of bronzes'. This part is one of that series and covers the determination of iron. The other parts are as follows:

- Part 1 Determination of copper and lead by electrolytic method (*first revision*)
- Part 2 Determination of manganese by photometric method (*first revision*)
- Part 3 Determination of phosphorus by volumetric method (*first revision*)
- Part 4 Determination of nickel by photometric method (*first revision*)
- Part 5 Determination of tin by iodimetric method (*first revision*)
- Part 6 Determination of zinc by complexometric (EDTA) method (*first revision*)
- Part 7 Determination of antimony by rhodamine B spectrophotometric method (*first revision*)

Methods of chemical analysis of other constituents in bronzes, namely, aluminium and silicon are under preparation and will be published in subsequent parts of this series.

In this revision determination of iron by potassium dichromate (volumetric) method has been updated for iron content above 0.5 percent. The determination of iron content below 0.5 percent by O-phenanthroline spectrophotometric method has been incorporated in the standard and has been brought up in line with ISO 1812 : 1976 'Copper and copper alloys — Determination of iron content — 1, 10-phenanthroline spectrophotometric method'.

In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated is to be rounded off, it shall be done in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'.

Indian Standard

METHODS OF CHEMICAL ANALYSIS OF BRONZES

PART 8 DETERMINATION OF IRON

(First Revision)

1 SCOPE

1.1 This standard (Part 8) covers methods for determination of iron in the range above 0.5 percent by potassium dichromate method and in the range below 0.5 percent by O-phenanthroline method in bronzes.

2 REFERENCES

2.1 The following Indian Standards are necessary adjuncts to this standard:

IS No.	Title
265 : 1987	Hydrochloric acid (<i>third revision</i>)
1817 : 1961	Methods for sampling non-ferrous metals for chemical analysis

3 SAMPLING

3.1 Samples shall be drawn and prepared in accordance with IS 1817 : 1961.

4 QUALITY OF REAGENTS

4.1 Unless specified otherwise, analytical grade reagents and distilled water shall be employed in the test.

5 DETERMINATION OF IRON (≥ 0.5 PERCENT) BY THE POTASSIUM DICHROMATE (VOLUMETRIC) METHOD

5.1 Outline of the Method

Iron is reduced in a boiling solution with stannous chloride, the excess of which is destroyed with mercuric chloride. The reduced iron is titrated with standard potassium dichromate solution using sodium diphenylamine sulphonate as indicator.

5.2 Reagents

5.2.1 *Dilute Nitric Acid* — 1 : 1 and 1 : 99 (v/v).

5.2.2 *Concentrated Nitric Acid* — rd = 1.42 (conforming to IS 265 : 1987).

5.2.3 *Perchloric Acid* — 70 percent (v/v).

5.2.4 *Hydrobromic Acid* — 48 percent (v/v).

5.2.5 *Dilute Ammonium Hydroxide* — 1 : 1 (v/v).

5.2.6 *Ammonium Chloride Solution* — 2 percent (m/v).

5.2.7 *Dilute Hydrochloric Acid* — 1 : 1 (v/v).

5.2.8 *Stannous Chloride Solution*

Dissolve by heating 60 g of pure stannous chloride in a mixture of 400 ml of concentrated hydrochloric acid and 600 ml of water until the solution is complete. Cool, add a few pieces of granulated tin and preserve the solution in an air-tight amber-coloured bottle to prevent oxidation.

5.2.9 *Mercuric Chloride Solution*

Prepare a saturated solution of mercuric chloride in water.

5.2.10 *Sulphuric Acid — Phosphoric Acid Mixture*

Add slowly 150 ml of concentrated sulphuric acid to 700 ml of water with continuous stirring. Add to this 150 ml of phosphoric acid. Destroy any oxidizable impurities by adding potassium permanganate solution (0.1 N) drop by drop until the pink colour of permanganate persists. Cool the solution.

5.2.11 *Sodium Diphenylamine Sulphonate Indicator Solution*

Dissolve 0.2 g of the reagent in 100 ml of hot water.

5.2.12 *Standard Potassium Dichromate Solution* (0.05 N).

Dry pure potassium dichromate in an oven at 105°C for one and a half hours. Cool in a desiccator and carefully transfer exactly 2.4517 g to a one-liter volumetric flask through a funnel. Wash the funnel thoroughly and dissolve the salt by shaking until it dissolves completely. Make up to the mark and mix well.

5.3 Procedure

5.3.1 Dissolve 5.00 g sample in 50 ml of dilute nitric acid (1 : 1) in a 250 ml beaker and heat to expel brown fumes. Dilute the solution to 100 ml and digest on a steam bath for one hour. Filter and wash with hot dilute nitric acid (1 : 99) and preserve the filtrate.

5.3.2 Transfer the paper to the original beaker. Add 15 ml of concentrated nitric acid and 10 ml of perchloric acid and heat gently until white fumes appear. Cool and wash down the sides of the beaker. Add 10 ml of hydrobromic acid and boil gently until white fumes appear. Repeat hydrobromic acid treatment to remove last traces of tin. Dilute and heat to dissolve the separated salts. Filter off any silica and combine the filtrate with the filtrate obtained in 5.3.1.

5.3.3 Adjust the volume of the solution to 150 ml and add dilute ammonium hydroxide (1 : 1) until faint smell of ammonia persists. Boil for a few minutes and allow to settle for some time. Filter while hot, using a medium-texture filter paper and wash (a few times alternately) with ammonical ammonium chloride solution and hot water. Repeat the precipitation of hydroxide once more following the same procedure as mentioned above. Combine the precipitate and dissolve through the filter paper with 20 ml of hot dilute hydrochloric acid (1 : 1) and wash thoroughly with hot water collecting the solution and washings in the original beaker.

5.3.4 Evaporate the solution to 15 to 20 ml. Heat to boiling and to the boiling solution add stannous chloride solution drop by drop with continuous stirring until the solution just becomes colourless. Add one or two drops excess of stannous chloride solution and wash down the sides with a little water. Cool the flask rapidly under running tap water until the contents have cooled down to room temperature.

5.3.5 Add excess of mercuric chloride solution (about 5 ml) in one instalment and shake the flask. At this stage a silky white precipitate appears. If it fails to appear or if a blackish precipitate appears, reject the test. If the precipitate is too much add further 5 ml of mercuric chloride solution. Keep for about two to five minutes and add 15 ml of sulphuric-phosphoric acid mixture and dilute to about 250 ml, washing down the sides of the flask. Add three to four drops of sodium diphenylamine sulphonate indicator solution and titrate slowly with standard potassium dichromate solution stirring continuously until the colour begins to darken. Continue the titration slowly till a drop of the dichromate solution produces a stable violet-blue colouration.

5.3.6 Make a blank determination following the procedure as specified in 5.3.1 to 5.3.5 and using the same amount of all reagents, but without the sample.

5.4 Calculation

$$\text{Iron, percent by mass} = \frac{(A - B) \times C}{D} \times 5.585$$

where

A = volume, in ml, of standard potassium dichromate solution required for test solution;

B = volume, in ml, of standard potassium dichromate solution required to titrate the blank;

C = normality of the standard potassium dichromate solution; and

D = mass, in g, of the sample taken.

6 DETERMINATION OF IRON (≤ 0.5 PERCENT) BY O-PHENANTHROLINE SPECTROPHOTOMETRIC METHOD

6.1 Outline of the Method

Iron from the test portion is extracted with methyl isobutylketone and complexed with O-phenanthroline and absorbance measured at 510 nm.

6.2 Reagents

6.2.1 Dilute Hydrochloric Acid — 2 : 1 and 1 : 1 (v/v).

6.2.2 Hydrogen Peroxide — 20 percent (v/v).

6.2.3 Methyl Isobutylketone

6.2.4 Ascorbic acid — 5 percent (w/v).

6.2.5 O-phenanthroline (Buffered)

Dissolve 1 g of reagent with 215 ml of glacial acetic acid and 265 ml of ammonia solution ($\rho_d = 0.90$). The pH of solution should be 6.5 ± 0.1 .

6.2.6 Standard Iron Solution

Dissolve 0.1 g of high purity iron in 20 ml of hydrochloric acid and dilute to 1 litre. Take 50 ml of this solution and dilute to 500 ml. The final solution contains 10 μg of iron per ml.

6.3 Procedure

6.3.1 Weigh 5.00 g of sample and dissolve in 40 ml of dilute hydrochloric acid (2 : 1). Add hydrogen peroxide in small quantity till the sample is dissolved completely. Cool, until the reaction ceases. Heat again to expel excess hydrogen peroxide and cool. Transfer the solution to 250 ml separating funnel and wash the beaker with dilute hydrochloric acid (1 : 1). Add 20 ml of methyl isobutylketone to the separating funnel and shake for 20 seconds. Allow the phases to separate, discard the aqueous phase and wash the organic phase by adding 5 ml portion of dilute hydrochloric acid (1 : 1) three times to make it free from copper.

6.3.2 Extract the iron from the organic layer with successive 10-ml portion of ascorbic acid. Transfer the aqueous extracts to 50 ml volumetric flask, mix with 5 ml buffered O-phenanthroline solution and make up. Measure the absorbance of solution at 510 nm against reagent blank.

6.3.3 Blank

Carry out a blank using same quantity of all reagents and following the same procedure as specified in 6.3.1 and 6.3.2 but without the sample.

6.3.4 Calibration Curve

Transfer 0, 5, 10, 15, 20 and 25 ml of standard iron solution in 50 ml volumetric flasks. To each of it add 20 ml of ascorbic acid and mix. Allow to stand for one minute and add 5 ml O-phenanthroline solution. Dilute to mark with water. Measure the absorbance at 510 nm and prepare a calibration curve.

NOTE — The zero number of the series of prepared

calibration solution (6.3.4) will serve as the blank for the calibration solution.

6.4 Calculation

Convert the spectrophotometric readings of two sample solutions to micrograms of iron by means of calibration curve. Calculate the percentage of iron as follows:

$$\text{Iron, percent by mass} = \frac{A}{B \times 10^6} \times 100$$

where

A = mass, in μg , of iron found in sample solution, and

B = mass, in g, of sample represented by aliquot taken.

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